Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

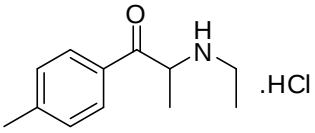
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D968: (±)-4'-Methylethylcathinone hydrochloride

Report ID: D968.2024.01

Chemical Formula: $C_{12}H_{17}NO.HCl$

Molecular Weight: 227.7 g/mol (HCl), 191.3 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
11-D-08	**1266688-86-1 (HCl)** **1225617-18-4 (base)**	**98.0 ± 1.8%**

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

IUPAC name: 2-(Ethylamino)-1-(4-methylphenyl)-1-propanone hydrochloride.

Expiration of certification: The property values are valid till 20 November 2034, ten years from the date of re-certification provided the **unopened** material is handled and stored in accordance with the recommendations below. The material as issued in the unopened container and stored as recommended below should be suitable for use beyond this date, subject to confirmation of batch stability from the issuing body. The expiry date/shelf life does not apply to sample bottles that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

Description: White powder prepared by synthesis and certified for identity and purity by NMI Australia. Packaged in amber glass bottles with a septum and crimped aluminium cap or screw top cap.

Intended use: This certified reference material is suitable for use as a primary calibrator.

Instructions for use: Equilibrate the bottled material to room temperature before opening.

Recommended storage: When not in use this material should be stored at or below 25 ºC in a closed container in a dry, dark area.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance all impurities are quantified as a mass fraction and subtracted from 100%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

Stability: This material has demonstrated stability over a minimum period of three years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. The long-term stability of the compound in solution has not been examined.

Homogeneity assessment: The homogeneity of the material was assessed using purity assay by GC-FID on eight randomly selected 1-2 mg sub samples of the material. The material was judged to be sufficiently homogeneous at this level of sampling as the variation in analysis results between samples was not significantly different at a 95% confidence level from that observed on repeat analysis of the same sample.

Safety: Treat as a hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust. Refer to the provided safety data sheet.

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
29 November 2024

This report supersedes any issued prior to 29 November 2024.

NATA Accreditation No. 198 / Corporate Site No. 14214.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100 % - I_{ORG}) x (100 % - I_{VOL} – I_{NVR}) Equation 1

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by qualitative elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800
	Column:	DB-17, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	80 °C, 10 °C/min to 280 °C (10 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the *N*-acetyl derivative:	
	Initial analysis:	Mean = 98.2%, s = 0.2% (8 sub samples in duplicate, November 2024)
GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	90 °C, 10 °C/min to 180 °C (3 min), 30 °C/min to 280 °C (10 min)
	Injector:	160 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the methyl carbamate derivative:	
	Initial analysis:	Mean = 98.5%, s = 0.1% (5 sub samples in duplicate, May 2013)
	Re-analysis:	Mean = 98.3%, s = 0.04% (10 sub samples in duplicate, June 2014)
	Re-analysis:	Mean = 98.4%, s = 0.07% (4 sub samples in duplicate, May 2017)
	Re-analysis:	Mean = 98.4%, s = 0.09% (5 sub samples in duplicate, April 2020)
GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	90 °C, 10 °C/min to 180 °C (3 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component *N*-acetyl derivative:	
	Initial analysis:	Mean = 98.1%, s = 0.1% (8 sub samples in duplicate, July 2011)
	Re-analysis:	Mean = 98.1%, s = 0.04% (5 sub samples in duplicate, May 2012)
GC-FID:	Instrument:	Varian CP-3800
	Column:	TG-17MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	180 °C, 5 °C/min to 240 °C (3 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the *N*-acetyl derivative:	
	Initial analysis:	Mean = 98.1%, s = 0.1% (8 sub samples in duplicate, July 2011)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (June 2011). The volatile content (e.g. organic solvents and/or water) could not be determined due to the volatility of the material.
Karl Fischer analysis:		Moisture content ~ 0.2% mass fraction (July 2011, May 2012 and 2014, April 2017 and 2020) Moisture content < 0.4% mass fraction (April 2013) Moisture content ~ 0.1% mass fraction (November 2024)

Spectroscopic and other characterisation data

GC-MS:	Free base:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	90 °C (1 min), 10 °C/min to 180 °C (7 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	20/1
	N-Acetyl derivative:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	90 °C (1 min), 10 °C/min to 180 °C (7 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	20/1
	The retention times of the free base and *N*-acetyl derivative are reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Free base (9.4 min):	119 (8), 91 (11), 72 (100), 70 (17), 44 (19), 42 (12) *m/z*
	N-Acetyl (14.4 min):	119 (9), 114 (63), 91 (11), 72 (100), 44 (9), 43 (7) *m/z*
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μm
	Program:	50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)
	Injector:	150 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	Ethanol, diethyl ether, ethyl chloride
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3060, 2976, 2928, 2794, 2739, 2696, 2477, 2373, 1689, 1606, 1458, 1440, 1307, 1241, 1189, 976, 832, 786, 737 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 1.36 (3H, t, *J* = 7.3 Hz), 1.59 (3H, d, *J* = 7.2 Hz), 2.42 (3H, s), 3.12 (1H, m), 3.26 (1H, m), 5.12 (1H, quartet, *J* = 7.2 Hz), 7.42 (2H, d, *J* = 8.0 Hz), 7.91 (2H, d, *J* = 8.3 Hz) ppm
		Ethanol estimated at 0.03% mass fraction was observed in the ^{1}H NMR. Diethyl ether and ethyl chloride were below the limit of detection.
^{13}C NMR:	Instrument:	Bruker Avance III-400
	Field strength:	101 MHz
	Solvent:	D_2O
	Spectral data:	δ 12.2, 17.2, 22.4, 42.8, 59.3, 130.5, 131.0, 131.3, 148.8, 198.5 ppm
Melting point:		217-218 °C
Microanalysis:	Found:	C = 63.5%; H = 8.2%; N = 6.2% (July 2011)
	Calculated:	C = 63.3%; H = 8.0%; N = 6.2% (Calculated for $C_{12}H_{17}NO.HCl$)